

COMPRESSION AND HARDNESS WITH FTIR CHARACTERIZATION OF UHMWPE NANOCOMPOSITES AS ACETABULAR CUP IN HIP JOINT REPLACEMENT

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ABSTRACT

Eight nano composites were prepared to use as an acetabular cup for hip joint implantation with a matrix of UHMWPE and nano-reinforcements include carbon nanotubes (CNTs) and nano hydroxyapatite (nHA) by adding four weight fractions involve (1, 2, 3 and 5%wt) of each material. FTIR spectra were used to identify the incorporation between the matrix and nano fillers and the shifting in main peaks confirmed the good cross linking that occurs within the composites structure.

Compression and hardness for prepared nanocomposites were investigated. The compressive strength was increased to get the highest values for UHMWPE/3%nHA followed by HUMWPE/3%CNTs composite due to nature of hydroxyapatite particles to fill the voids compared with the structure of nanotubes. The improving of hardness also observed at 3% of nanoadditives, but UHMWPE/3%CNTs composite has a hardness higher than HUMWPE/3%nHA because of the structure of tubes that act as fibers to get hard material compared with reinforcing by particles of HA.

KEYWORD: Hip Joint Replacement, Acetabular Cup, Nanocomposite; UHMWPE, Nano Additives, Hardness, Compression & Toughness

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INTRODUCTION

Ultrahigh molecular weight polyethylene (UHMWPE) represents as standard material to use in bio applications due to superior wear resistance along with high fracture toughness and biocompatibility compared to other polymers [1]. Due to the properties of UHMWPE like wear, rigidity therefore, has been preferred as the matrix and represents an alternative material to HDPE in bio applications[2], therefore it is successfully used in biomedical especially in articulated joint implants[3]. Its application in the human body is associated with its biocompatibility, inert strength and tribological properties [4, 5]. Attempts have been made to improve the properties of polymeric composites by reinforcing with other materials especially by dispersion with nano-fillers for biomedical and structural applications [6-8]. In addition to that, the nano-reinforcing materials enhance the properties of the interphase of nanocomposites [9, 10]

The most application of UHMWPE is in hip joint as acetabular cup[11]. The parts of the hip prosthesis are shown in Figure (1).

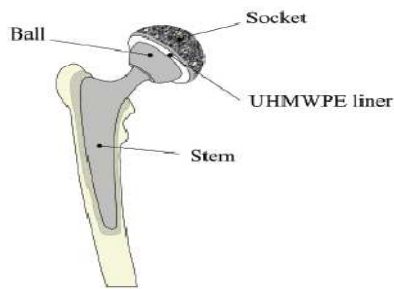


Figure 1: Typical Hip Prosthesis [11]

The inorganic fillers used as a reinforced with UHMWPE in order to improve the mechanical, physical and biological properties of UHMWPE [12-14]. Different types of carbon materials (fibers, particles, and nanotubes), have been used as a filler in UHMWPE because of good thermal and tribological properties[15- 17], but these additives must be in a small amount to achieve low cost with satisfactory properties of composites, which lead to the application in articulated joints. Therefore, the cost and properties to high performance are a great important point in the further study. In recent research nanocomposite, consist of UHMWPE as matrix reinforced with CNTs and nHA were investigated as an acetabular cup for hip joint by testing some mechanical properties.

EXPERIMENTAL PROCEDURE

Materials and Methods

Ultra-high molecular weight polyethylene (UHMWPE) from (MAX PIPE INDUSTRY Co. Ltd) with an average molecular weight (5.5×10^6 g/mol), density (0.935 g/cm^3) and average particle size of (20-50 μm) was used as the matrix to prepared eight nanocomposites. These nanocomposites were reinforced with four weight fraction (%) of each carbon nanotubes (CNTs) and nanohydroxyapatites (nHA).

Multi-walled carbon nanotubes (MWCNTs) (>95%, with an average diameter(30-80 nm) and lengths of 10 - 30 μm) were syntheses via AAO templates as describes in our previous paper [18], while nanohydroxyapatite (nHA) was supplied as a nano-particles from (Merck, Darmstad, Germany Company)with average particle size (80 nm).

Four weight fractions include 1, 2, 3 and 5% of each nanoadditives to produce nanocomposites that prepared by weighing of chosen nanofillers (CNTs and nHA) to reinforce polymer matrix UHMWPE and mixed with 30 mL of ethanol and then stirred the mixture by a hot magnetic stirrer for 45 min. and 60 °C to disperse the additive in solution. The final mixture (Ethanol + Additive + UHMWPE) was put in siliphon paper and input inside the dry oven for 20 min. at 60 °C, after drying it left in atmospheric for 72 hrs. to evaporate the residual ethanol.

The hydraulic hot press was used to fabricate UHMWPE nanocomposites. After the previous steps, the final produced mixture was put in a hot plate of the hydrulic press with a temperature range of (195-200 °C) and then pressed under 12 MPa. for 1.5 hrs at polymer Department inmaterialsengineeringcollege, Babylon unversity. Cooling the molds were done in the air to room temperature to get specimens and then they cut by CNC laser machine according to international standard specifications for each test in this research which agreement with ASTM standard.

Properties Measurement

Compression Test

The compression test is achieved according to (ASTM D695-02a) using the tensile machine at across head (strain rate) of (5mm/min) and the compressive load was applied gradually until fracture of the specimen occurs [19-21]. Figure (2) represent the specimens of compression test according to ASTM.

Hardness Test

Hardness test type (Shore-D) was carried out on UHMWPE nanocomposites. The dimensions of specimens were (40 mm in diameter and 4 mm in thickness). The hardness test was achieved according to (ASTM D2240) by durometer hardness test, type (Shore D) at load applied equally to 50 N and depressing time of measuring equal to (15 sec). The surface of specimens requirement to be very smooth in zone testing. The hardness value is very sensitive to the (specimen thickness, specimen diameter, and distance from the edge more than 12 mm). Therefore, the minimum thickness of the specimen is (3 mm) with a diameter more than (30mm). All specimens were tested five times at different sites for each specimen at the same time and an average value was taken [22, 23]. Figure (3) represent the specimens of Hardness test according to ASTM.

FTIR Spectroscopy

The (FTIR) test was achieved according to (ASTM E1252) using Fourier transform infrared spectrometer (FTIR), the origin from (Bruker Optics Company), type (SENSOR-27). FTIR test carried out in the air after placed the specimen inside the device, Fourier transform analysis were performed for pure UHMWPE, CNTs, and nHAAs well as UHMWPE nanocomposites reinforced by (CNTs) and (nHA) nanoparticles. Infrared spectrums were obtained in absorption and were set to operate in the range of ($400 - 4000 \text{ cm}^{-1}$) at the thickness of specimens between the (4 mm) as rectangular rod form with cross-sectional area equal to (0.16 mm^2) [24].

RESULTS AND DISCUSSIONS

Characterization of Nanocomposites by FTIR Spectroscopy

FTIR of UHMWPE is shown in Figure (4) which indicates the important bands assigned at (729.91 cm^{-1}) referred to $=\text{CH}_2$ out-of-plane bending, and at 1462.16 cm^{-1} for C–H bending deformation. The final two sharp high-intensity peaks at 2915.79 and 2848.13 cm^{-1} are characterized by the $-\text{CH}_2$ asymmetric stretching and symmetric stretching, respectively. While Figure (5) shows the FTIR spectrum of UHMWPE in permeability, this spectrum indicates C–H stretching occurs around 3000 cm^{-1} , CH_2 methylene groups have a characteristic absorption of approximately 1465 cm^{-1} , and C–C stretching is not interpretatively useful (it has many peaks).

Figures (6) and (7) show the FTIR spectra for pure CNT and HA respectively. The spectrum of CNT indicates the two dominant peaks; 1649 and 3468 cm^{-1} (associated with O–H). Several peaks in the range of 3000 cm^{-1} are attributed to CH_x groups. The vibration at near 1593 cm^{-1} is an indication to the presence of the cylinder like carbon structure (rolled graphene sheet). Several bands at near 1580 cm^{-1} are referring to active modes in the infrared spectrum and depending on the geometry of the CNT and nanotube diameter as observed by Dyke and Tour[25]. In addition to appearing the stretching of C=C bond around 1650 cm^{-1} .

The spectrum of HA in Figure (7) shows the characteristic peaks appeared at 1048 cm^{-1} and 564.09 cm^{-1} that are assigned to the phosphate groups of HA. The broad stretching band at 3443.18 cm^{-1} and 603.11 cm^{-1} are assigned to the stretching and bending vibration of OH^- groups of HA, respectively.

The spectra of UHMWPE/CNTs nanocomposites are shown in Figure (8), this figure indicates the main peaks in UHMWPE such as stretching of C–H, CH_2 groups and C–C bonds with some shifting due to some bonding with additives. C–H stretching became clearer in composites because of the presence of CNTs that contain more C–H bond. Also, this clearance occurs in CH_2 groups due to conjugation effect. On the other hand, there are two dominant peaks in the spectrum at 1652 and 3483 cm^{-1} which attributed to functionalized CNT and another peak at 1472 cm^{-1} . The peak presented at $\approx 1450\text{ cm}^{-1}$ is a unique to MWCNTs as referred by Misra et al. [26]. The broader band at $\approx 3400\text{ cm}^{-1}$ is attributed to COOH groups by functionalization [27].

Figure (9) shows the spectra of UHMWPE/nHA nanocomposites, this figure indicates the presence of main peaks in pure UHMWPE with decreasing in vibration bands for pure HA due to agglomeration of HA particles within the chains of UHMWPE.

Compression of UHMWPE Nanocomposites

Figure (10) shows the relationship between the weight fraction of additives (CNTs or nHA) in a polymer matrix (UHMWPE) and compression strength of nanocomposites. This figure indicates the increasing of compression strength with increasing of the weight fraction of both additives (CNTs and nHA) up to 3% and then there is a little decreasing in compression values with adding 5%. Generally, the presence of nHA as reinforcement gave more compression strength than CNTs due to the different structure of both nanomaterials, the hollow structure of CNTs compared with nHA crystals (Hexagonal system) allows to nanocomposite to be more liable to compression than that reinforced with nHA. But the increase in compression strength for composites is due to the filling the voids within polymeric composite by reinforced particles by cross-linking with matrix [28, 29]. The compression strength reaches to 40.5 MPa. and 28.5 MPa. for UHMWPE/3% nHA and UHMWPE/3% CNTs compared with 20 MPa. for pure UHMWPE. After that, there are decreasing in compression strength due to the agglomeration for CNTs at 5% [30, 31]

Hardness of UHMWPE Nanocomposites

Figure (11) shows the relationship between the weight fraction of (CNTs and nHA) and the hardness (Shore D) of the nanocomposites, the behavior is similar to the that observed in compression which is confirm the improvement of mechanical properties by reinforcing with nanoparticles up to certain concentration (3%). It is known that the nanomaterials have unique hardness compared with micro-sized materials, and then the hardness will increase for materials reinforced by nanomaterials. In Figure (9), can be seen that the increase of hardness in the presence of CNTs is higher than in the presence of nHA due to the pure mechanical properties of each additive compared with pure UHMWPE. The largest values of hardness were observed for (UHMWPE/3% CNTs) which equal to (66 Shore D), followed by (65.5 shore D) for (UHMWPE/3% nHA) nanocomposite compared with a hardness of pure UHMWPE (61.6 Shore D). HA has a vital role to increase the hardness and roughness of almost materials when it's added to them [32]. After that, there are decreasing in hardness due to the agglomeration for nanofillers at 5% [33, 34].

CONCLUSIONS

The experimental results of UHMWPE biocomposite indicated the following conclusions:

- Good agreement or correlation in FTIR spectrum between Pure UHMWPE specimen with the biocomposite specimens that reinforced by nHA and CNTs powders.
- Pure UHMWPE had the lowest value of compression strength and hardness which is equal to (20 MPa. & 61.6 Shore D) respectively.
- The highest value of compressive strength was (40.5 MPa.) happened for UHMWPE/3%nHA.
- The highest value of hardness was (66 shore D) happened for UHMWPE/3%CNTs.

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APPENDICES



UHMWPE/nHAUHMWPE/CNTs

Figure 2: Specimens to Compression Test



UHMWPE/nHAUHMWPE/CNTs

Figure 3: Specimens to Hardness Test

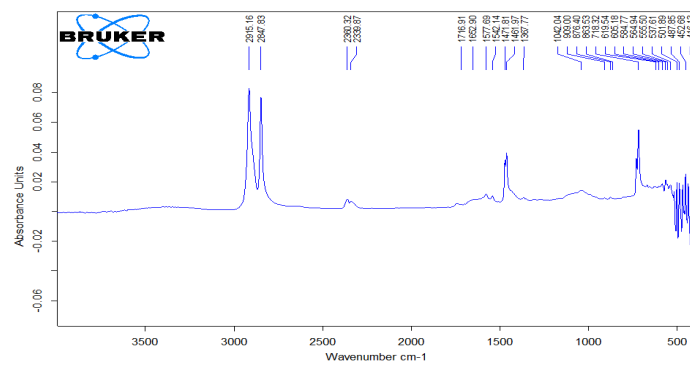


Figure 4: FTIR Analysis of Pure UHMWPE

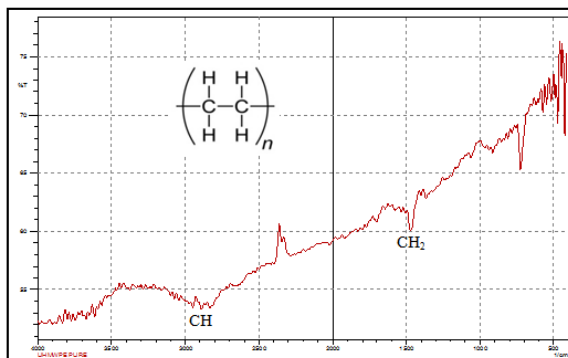


Figure 5: FTIR Spectrum of Pure UHMWPE Permeability

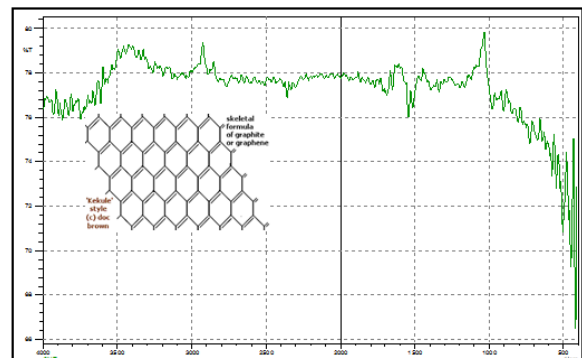


Figure 6: FTIR Spectrum of CNT

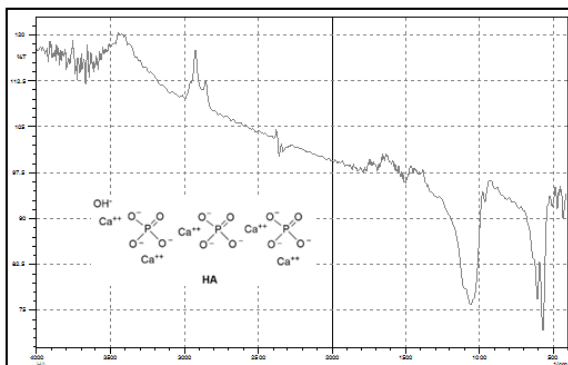


Figure 7: FTIR Spectrum of HA

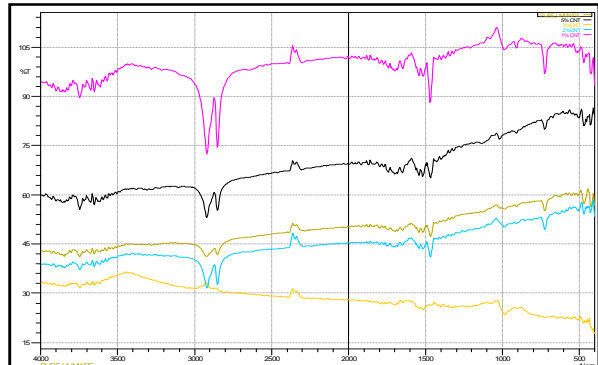


Figure 8: FTIR Spectrum of UHMWPE/CNTs Nanocomposites

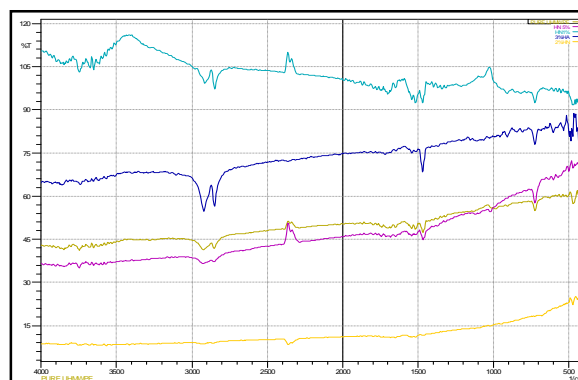


Figure 9: FTIR Spectrum of HA/UHMWPE Nanocomposites

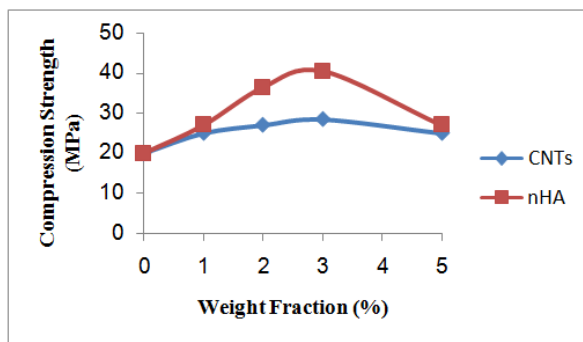


Figure 10: Compression Strength of UHMWPE Nanocomposites with Different Additives

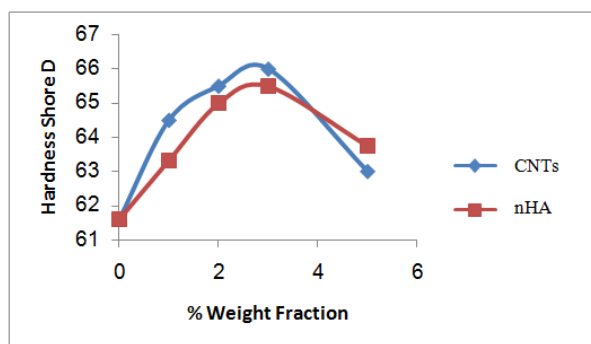


Figure 11: Hardness for Nanocomposites with Different Additives

